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Limiting Molar Conductances and Thermodynamic Association Constants for Nickel(II), Cadmium(II), Magnesium(II), and Copper(II) Sulfates in Mixtures of Methanol and Water at 293.15 K

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Electrolytic conductivities of dilute solutions of nickel(II), cadmium(II), magnesium(II), and copper(II) sulfates in binary mixtures of methanol and water have been measured at 293.15 K. The limiting molar conductances (Λ_0) and the ion-association constants (K_A) of the electrolytes have been evaluated by analysis of the conductance data using the Lee and Wheaton conductivity equation in the Pethybridge and Taba modification.

Introduction

The study of transport properties (conductance, viscosity, transference numbers) of electrolytes is important because the values provide information about the solvation behavior of the ions. The use of mixed solvents makes possible the variation of the dielectric constant, so the ion solvation effect can be better studied. Solutions of 2:2 salts in water are electrostatically equivalent to 1:1 salts in a solvent of low dielectric constant (∼20), implying that an unassociated 2:2 electrolyte is essentially impossible.[1,2]

In the literature appear reports of conductivity measurements of transition-metal (II) sulfates in mixed solvents such as $ZnSO_4$ and $CuSO_4$ in water with dioxan, ethanol, acetone, and ethylene glycol;[3] $MnSO_4$ in methanol + water,[4] in dioxane + water,[5] in acetone + water,[6] in ethylene glycol + water;[7] $MgSO_4$ in ethanol + water;[8] and $CuSO_4$ and $MnSO_4$ in ethanol + water.[9]

As a part of an extensive investigation on the transport properties of 2:2 electrolytes in mixed solvents, we report conductance measurements of $NiSO_4$, $CdSO_4$, $MgSO_4$, and $CuSO_4$ in binary mixtures of methanol + water and in a composition range from 0 to 80% mass of methanol at 293.15 K. The concentration range in which conductance measurements were made was 0.0001−0.0060 mol·dm^{-3}. The limiting molar conductivities (Λ_0) and the association constants (K_A) of the electrolytes have been derived. The results indicate the presence of specific ion−solvent and ion−ion forces in the solutions. These are reflected strongly in the values of Λ_0 and K_A.

Experimental Section

Methanol (Merck, 99.5%) with density 0.791 45 g·cm^{-3}, viscosity 0.587 mPa·s, and refractive index 1.3294 at 293.15 K was used for the present measurements without further purification. These physical constants are in agreement with the literature values, $\rho = 0.791\ 29$ g·cm^{-3}, $\eta = 0.5929$ mPa·s, and $n_D = 1.3284$.[10] The water was doubly distilled, and the conductance was found to be better than 0.50 μS at 293.15 K.

Nickel sulfate hexahydrate ($NiSO_4 \cdot 6H_2O$) (Merck, 99.0%), cadmium sulfate monohydrate ($CdSO_4 \cdot H_2O$) (Fluka, 99.0%), magnesium sulfate anhydrous ($MgSO_4$) (Sigma, 99.5%), and copper sulfate anhydrous ($CuSO_4$) (Fluka, 99.5%) were used without further purification.

Table 1. Densities (ρ), Viscosities (η), and Specific Conductivities (κ) of Methanol (1) + Water (2) Mixtures at 293.15 K

x_1	ρ / g·cm^{-3}	ς / mPa·s	$10^6\kappa$ / S·cm^{-1}
0.0000	0.998 22	1.002	0.55
0.0588	0.981 27	1.318	0.76
0.1233	0.966 48	1.618	0.70
0.1942	0.951 59	1.805	0.53
0.2727	0.934 71	1.850	0.52
0.4576	0.894 32	1.612	0.51
0.6923	0.846 57	1.126	0.40
1.0000	0.791 45	0.587	0.15

The methanol + water mixtures were prepared by mass (±0.0001 g). The mole fractions were known to ±0.0001 in all cases. A stock solution for each salt in pure water or the appropriate methanol + water mixture was prepared by mass, and the solutions were prepared by dilution. Molar concentrations of the solutions were obtained from the salt and solution masses and the density values. The accuracy of the molar concentrations was ±0.000 01 mol·dm^{-3}.

Densities were measured with an Anton Paar (DMA 58) microcomputer-controlled precision densimeter with a built-in solid-state thermostat at (293.15 ± 0.01) K. The estimated uncertainty of the measured densities was ±0.000 01 g·cm^{-3}. Flow times of the solvent mixtures were measured with a viscosity measuring unit (Schott Geräte AVS 310), equipped with an Übbelohde capillary viscometer. The temperature was maintained constant within ±0.03 K. The accuracy in the viscosity measurements was ±0.001 mPa·s. The measuring procedures for density and viscosity are described in previous papers.[11,12]

The conductance measurements were carried out using a digital bridge-type conductivity meter (Jenway, PCM 3) working at a frequency of 1 kHz. The accuracy was ±0.5%. A dipping type conductance cell with platinized electrodes was used. The cell constant (0.98 ± 0.01 cm^{-1}) was determined by calibration with a 0.01 mol·dm^{-3} aqueous KCl solution at (293.15 ± 0.01) K. The temperature of each solution became stable within 10−15 min and remained

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Table 2. Densities (ρ) and Molar Conductivities (Λ) for Copper(II), Magnesium(II), Cadmium(II), and Nickel(II) Sulfates in Methanol (1) + Water (2) Mixtures at 293.15 K

$10^2/$ mol·dm^{-3}	ρ g·cm^{-3}	Λ S·cm^2·mol^{-1}	10^2c mol·dm^{-3}	ρ g·cm^{-3}	Λ S·cm^2·mol^{-1}	$10^2/$ mol·dm^{-3}	ρ g·cm^{-3}	Λ S·cm^2·mol^{-1}	10^2c mol·dm^{-3}	ρ g·cm^{-3}	Λ S·cm^2·mol^{-1}
Copper(II) Sulfate											
$x_1 = 0.0000$						$x_1 = 0.1942$					
0.016	0.998 22	221.19	0.064	0.998 29	199.80	0.026	0.951 59	101.65	0.069	0.951 66	88.04
0.028	0.998 23	214.85	0.082	0.998 32	194.91	0.038	0.951 60	97.90	0.079	0.951 66	85.41
0.045	0.998 26	207.30	0.090	0.998 34	193.58	0.049	0.951 60	93.54	0.092	0.951 74	83.13
0.060	0.998 29	201.23	0.109	0.998 37	188.00	0.059	0.951 65	90.97	0.106	0.951 74	81.27
$x_1 = 0.0588$						$x_1 = 0.2727$					
0.011	0.981 42	149.30	0.061	0.981 50	128.36	0.011	0.934 65	94.95	0.053	0.934 68	72.31
0.020	0.981 44	142.63	0.069	0.981 50	125.94	0.012	0.934 65	94.62	0.064	0.934 70	68.73
0.040	0.981 47	133.09	0.078	0.981 51	124.24	0.026	0.934 66	83.94	0.075	0.934 71	66.46
0.050	0.981 49	131.09	0.088	0.981 52	122.77	0.041	0.934 67	76.60	0.086	0.934 72	64.08
$x_1 = 0.1233$						$x_1 = 0.4576$					
0.022	0.966 60	128.78	0.062	0.966 67	113.27	0.013	0.894 60	55.88	0.056	0.894 62	39.89
0.033	0.966 63	124.97	0.075	0.966 71	110.13	0.022	0.894 61	50.74	0.063	0.894 63	37.84
0.042	0.966 64	120.71	0.090	0.966 73	106.93	0.038	0.894 61	43.41	0.072	0.894 64	36.71
0.054	0.966 64	115.84	0.110	0.966 73	101.91	0.045	0.894 62	42.14	0.082	0.894 65	33.40
Nickel(II) Sulfate											
$x_1 = 0.0000$						$x_1 = 0.2727$					
0.025	0.998 23	227.36	0.074	0.998 31	213.65	0.011	0.934 63	110.73	0.057	0.934 81	
0.037	0.998 25	223.02	0.087	0.998 33	210.84	0.026	0.934 73	99.83	0.068	0.934 81	82.59
0.050	0.998 27	218.88	0.098	0.998 35	210.16	0.036	0.934 75	94.35	0.083	0.934 81	78.69
0.062	0.998 30	216.59	0.125	0.998 39	203.85	0.046	0.934 79	91.16	0.091	0.934 82	76.44
$x_1 = 0.0588$						$x_1 = 0.4576$					
0.013	0.981 39	194.13	0.064	0.981 49	169.54	0.008	0.894 60	90.12	0.040	0.894 63	61.58
0.024	0.981 42	184.97	0.075	0.981 49	165.95	0.013	0.894 61	81.98	0.056	0.894 63	54.77
0.041	0.981 42	176.37	0.085	0.981 50	163.36	0.020	0.894 61	73.75	0.063	0.894 64	53.12
0.053	0.981 45	174.65	0.095	0.981 50	160.58	0.032	0.894 63	66.69	0.069	0.894 70	51.13
$x_1 = 0.1233$						$x_1 = 0.6923$					
0.016	0.966 60	147.16	0.070	0.966 77	125.00	0.010	0.847 02	41.52	0.056	0.847 06	24.40
0.033	0.966 69	137.87	0.083	0.966 77	121.59	0.020	0.847 04	36.61	0.071	0.847 07	22.31
0.045	0.966 71	132.91	0.096	0.966 81	119.21	0.030	0.847 05	31.49	0.081	0.847 09	22.01
0.057	0.966 75	130.00	0.110	0.966 82	116.26	0.040	0.847 06	28.01	0.091	0.847 09	20.04
$x_1 = 0.1942$											
0.012	0.951 48	127.07	0.057	0.951 54	105.31						
0.022	0.951 52	122.35	0.072	0.951 55	100.20						
0.036	0.951 53	113.90	0.091	0.951 58	95.75						
0.046	0.951 53	109.84	0.101	0.951 58	93.57						
Cadmium(II) Sulfate											
$x_1 = 0.0000$						$x_1 = 0.2727$					
0.038	0.998 32	192.65	0.140	0.998 51	167.48	0.022	0.934 84	73.75	0.267	0.935 28	44.92
0.057	0.998 36	188.48	0.150	0.998 54	162.88	0.055	0.934 93	69.03	0.347	0.935 35	41.35
0.079	0.998 40	181.54	0.195	0.998 61	160.70	0.131	0.934 94	55.53	0.429	0.935 45	38.43
0.110	0.998 46	177.87				0.182	0.935 15	50.21			
$x_1 = 0.0588$						$x_1 = 0.4576$					
0.024	0.981 42	153.31	0.193	0.981 80	120.88	0.012	0.895 01	58.72	0.061	0.895 20	39.86
0.068	0.981 56	138.62	0.218	0.981 85	116.11	0.025	0.895 07	53.15	0.082	0.895 20	35.38
0.093	0.981 63	132.96	0.246	0.981 90	110.92	0.034	0.895 19	49.18	0.102	0.895 32	31.85
0.146	0.981 73	126.15				0.044	0.895 19	45.08			
$x_1 = 0.1233$						$x_1 = 0.6923$					
0.059	0.966 86	107.62	0.260	0.967 35	74.39	0.008	0.846 88	31.78	0.025	0.846 91	22.11
0.104	0.966 92	97.60	0.308	0.967 39	72.17	0.013	0.846 89	28.68	0.042	0.846 94	18.11
0.160	0.967 16	90.25	0.354	0.967 52	71.13	0.019	0.846 89	24.78	0.044	0.846 95	17.55
0.205	0.967 25	84.86				0.022	0.846 90	23.41			
$x_1 = 0.1942$											
0.052	0.951 61	85.14	0.409	0.952 42	54.27						
0.076	0.951 69	81.37	0.545	0.955 26	47.92						
0.154	0.951 88	72.86	0.629	0.952 66	43.08						
0.269	0.952 11	62.84									
Magnesium(II) Sulfate											
$x_1 = 0.0000$						$x_1 = 0.2727$					
0.029	0.998 30	216.48	0.204	0.998 52	177.22	0.020	0.934 88	101.00	0.070	0.935 15	81.52
0.064	0.998 35	203.43	0.253	0.998 58	169.84	0.027	0.935 02	95.73	0.079	0.935 15	79.19
0.116	0.998 41	192.62	0.294	0.998 63	169.93	0.040	0.935 03	90.72	0.082	0.935 16	78.38
0.158	0.998 46	185.40				0.050	0.935 03	87.15	0.104	0.935 17	74.08
$x_1 = 0.0588$						$x_1 = 0.4576$					
0.035	0.981 55	169.91	0.203	0.981 85	139.40	0.028	0.894 77	65.25	0.326	0.895 11	33.50
0.054	0.981 66	163.19	0.270	0.981 86	130.12	0.054	0.894 95	60.65	0.416	0.895 59	30.85
0.095	0.981 67	152.11	0.332	0.981 86	126.62	0.103	0.894 89	50.77	0.519	0.895 68	28.52
0.138	0.981 72	148.37				0.200	0.895 11	41.13			
$x_1 = 0.1233$						$x_1 = 0.6923$					
0.072	0.966 95	120.76	0.229	0.967 15	97.24	0.038	0.847 40	33.07	0.064	0.847 42	27.14
0.114	0.966 96	113.70	0.302	0.967 19	89.98	0.040	0.847 40	33.05	0.075	0.847 42	25.56
0.138	0.967 05	110.95	0.382	0.967 30	83.91	0.052	0.847 41	29.67	0.086	0.847 43	24.09
0.183	0.967 15	100.09				0.063	0.847 41	26.68	0.092	0.847 43	22.51
$x_1 = 0.1942$											
0.052	0.951 51	103.14	0.280	0.951 73	72.29						
0.116	0.951 55	89.83	0.373	0.951 90	64.81						
0.170	0.951 65	83.55	0.428	0.951 88	63.24						
0.212	0.951 68	79.93									

Table 3. Limiting Molar Conductances (Λ_0) and Association Constants (K_A) for Copper(II), Magnesium(II), Cadmium(II), and Nickel(II) Sulfates in Methanol (1) + Water (2) Mixtures at 293.15 K

x_1	Λ_0 $S\cdot cm^2\cdot mol^{-1}$	K_A $dm^3\cdot mol^{-1}$	σ_Λ
	Copper(II) Sulfate		
0.0000	229.3 ± 0.9 266.16[b]	253 ± 1 188[b]	0.321
0.0588	154.2 ± 0.8	396 ± 1	0.323
0.1233	142.5 ± 0.7	512 ± 1	0.105
0.1942	116.1 ± 0.6	609 ± 1	0.074
0.2727	108.0 ± 0.6	1403 ± 3	0.025
0.4576	73.2 ± 0.4	2978 ± 6	0.022
0.6923	*a*	*a*	
	Nickel(II) Sulfate		
0.0000	234.4 ± 0.9	139 ± 1	0.558
0.0588	201.0 ± 0.9	342 ± 1	0.408
0.1233	156.2 ± 0.8	437 ± 1	0.155
0.1942	138.9 ± 0.7	753 ± 2	0.121
0.2727	122.7 ± 0.6	1078 ± 2	0.041
0.4576	112.2 ± 0.6	3856 ± 8	0.015
0.6923	66.2 ± 0.3	8281 ± 17	0.011
	Cadmium(II) Sulfate		
0.0000	209.9 ± 0.9	222 ± 1	1.137
0.0588	161.9 ± 0.8	262 ± 1	0.657
0.1233	138.4 ± 0.7	564 ± 1	0.375
0.1942	107.2 ± 0.6	525 ± 1	0.446
0.2727	84.5 ± 0.4	612 ± 1	0.222
0.4576	79.1 ± 0.4	3212 ± 6	0.044
0.6923	58.5 ± 0.3	17731 ± 35	0.030
	Magnesium(II) Sulfate		
0.0000	225.2 ± 0.9	164 ± 1 167.7[c] 210[d]	1.264
0.0588	179.3 ± 0.9	189 ± 1	0.917
0.1233	149.7 ± 0.8	379 ± 1	0.415
0.1942	125.0 ± 0.6	467 ± 1	0.262
0.2727	115.3 ± 0.6	858 ± 2	0.055
0.4576	85.4 ± 0.4	1226 ± 2	0.110
0.6923	59.9 ± 0.3	4368 ± 9	0.021

[a] Copper sulfate was not sufficiently soluble in the methanol rich region of the mixtures. [b] At 298.15 K (Niazi and Hussain, 1994).[9] [c] At 298.15 K (Pethybridge and Taba, 1977).[2] [d] At 298.15 K (Quintana et al., 1986).[8]

constant. All data were corrected with the specific conductance of the solvent. The accuracy of the molar conductances was ±0.5%.

In all cases the measurements were repeated twice with different stock solutions to obtain reproducible results.

The dielectric constants of the binary and of the pure solvents were taken from the literature.[13,14]

Results and Discussion

Densities, viscosities, and specific conductivities of pure water and methanol + water mixtures are reported in Table 1. The viscosity and specific conductance values show a significant change (maximum) with the change in the solvent composition of the mixed solvent, indicating that the methanol + water mixture exhibits nonideal behavior. The addition of methanol to water promotes a breakdown of the water structure so the oxygen centers of the water molecules become free for interaction with the protons of methanol molecules.[15]

The molar conductances of each electrolyte in the methanol + water mixtures are given in Table 2.

The experimental data were analyzed with the Lee and Wheaton[16, 17] conductivity equation in the form suggested by Pethybridge and Taba,[18] and the following set of equations was used

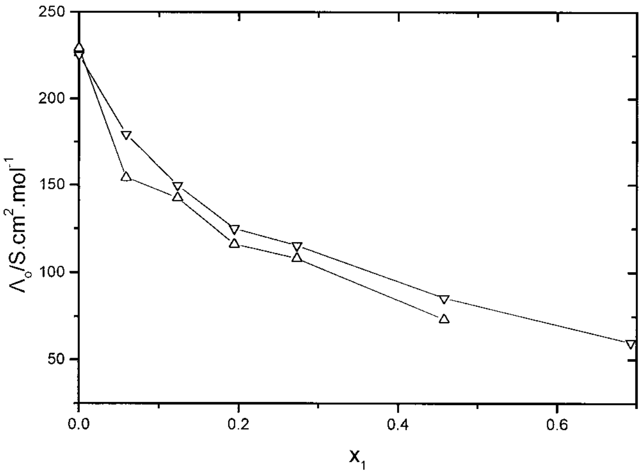

Figure 1. Variation of limiting molar conductivities with mole fraction for $CuSO_4$ (△) and $MgSO_4$ (▽) in methanol (1) + water (2) mixtures at 293.15 K.

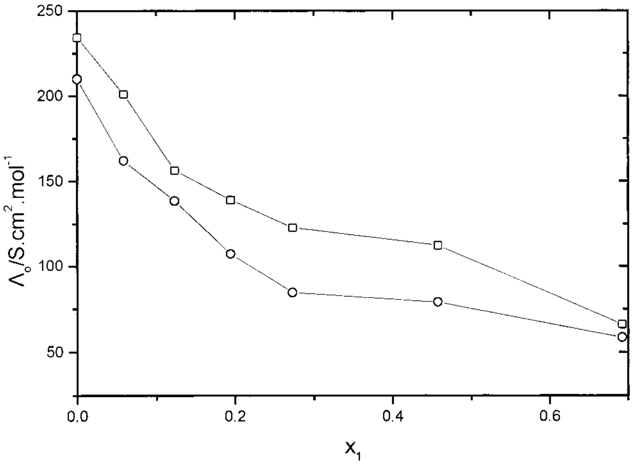

Figure 2. Variation of limiting molar conductivities with mole fraction for $CdSO_4$ (○) and $NiSO_4$ (□) in methanol (1) + water (2) mixtures at 293.15 K.

$$\Lambda = \gamma\left[\Lambda_0\left(1 + \frac{\Delta x}{x}\right) - \Delta\Lambda_e\right] \tag{1}$$

$$K_A = \frac{(1-\gamma)}{\gamma^2 f_\pm{}^2 c} \tag{2}$$

$$-\ln f_\pm = \frac{\beta\kappa}{2(1+\kappa R)} \tag{3}$$

where $\Delta x/x$ and $\Delta\Lambda_e$ are the relaxation and electrophoretic terms, respectively, β is twice the Bjerrum's distance, and κ^{-1} is the Debye length. The values of R were actually equal to Bjerrum's critical distance, a procedure which is mentioned by other researchers.[19−23] The other symbols have their usual meanings.

The Λ_0 and K_A values were obtained as the best fit parameters which minimize the standard deviation

$$\sigma_\Lambda{}^2 = \frac{\sum(\Lambda_{cald} - \Lambda_{obsd})^2}{(n-2)} \tag{4}$$

The values of Λ_0 and K_A for $NiSO_4$, $CdSO_4$, $MgSO_4$, and $CuSO_4$ in methanol + water mixtures, together with σ_Λ,

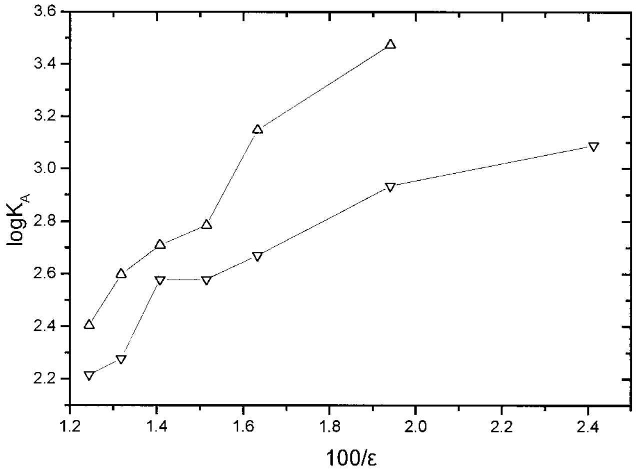

Figure 3. Variation of association constants with the inverse dielectric constant for $CuSO_4$ (△) and $MgSO_4$ (▽) in methanol (1) + water (2) mixtures at 293.15 K.

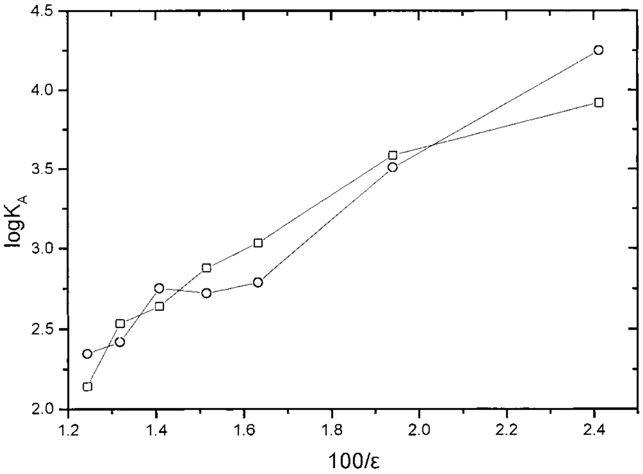

Figure 4. Variation of association constants with the inverse dielectric constant for $CdSO_4$ (○) and $NiSO_4$ (□) in methanol (1) + water (2) mixtures at 293.15 K.

are summarized in Table 3. The values given in the literature are also listed in Table 3.

The values of Λ_0 for the electrolytes in methanol + water mixtures are represented graphically in Figures 1 and 2 and the dependence of the log K_A values on the inverse of the dielectric constant of the binary solvent is represented in Figures 3 and 4.

The limiting molar conductivities decrease rapidly, and the association constants increase, as the methanol content of the mixture increases. The values of the association constants show that these salts do not exist in a completely dissociated form even in the aqueous solutions. The bivalent cations form ion pairs and water-separated pairs with the sulfate ions. As the methanol content is increased (dielectric constant of the solvent mixture decreases), the hydration becomes less strong and a short range interaction is possible. Therefore, it can be clearly considered that electrostatic ion pairs are formed. An examination of molecular models suggests that when methanol solvates the ions, perhaps a steric hindrance occurs.[24] Because of this steric hindrance, the oxygen atoms of methanol are not in direct contact with the ions; consequently, the ions move freely and they have a tendency to associate through ion−ion interactions.

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